

APPENDIX A

1. PURPOSE

The following measurements were performed to specify a structure of a denatured polyvinyl alcohol described in the Examples of the present invention.

2. ANALYTICAL METHOD

2-1. SAMPLES AND PROCESS

Prepared samples were dissolved in dense dimethylsulfoxide (70°C) for a ^{13}C -NMR(BCM) measurement and also for a ^{13}C -NMR(DEPT) measurement. Methanol was added by about 0.5% to a part of the samples before the ^{13}C -NMR(BCM) measurement, since the samples might contain methanol as one of the original components.

2-2. ANALYZER

FT-NMR apparatus: LA400 (JEOL)

2-3. MEASUREMENT CONDITIONS

Concentration: about 10%

BCM: proton complete decoupling measurement

DEPT: 135° (methine and methyl face upward while methylene faces downward; quaternary carbon disappears)

Conditions for the measuring apparatus are described in the charts.

3. RESULTS AND CONSIDERATION

- 1) Chart No. 1 shows results of ^{13}C -NMR measurement on the samples.

In the measurement, PVA as a main component was recognized together with polyvinyl acetate (VAC) and 2-substitutional benzene. The VAC was regarded as an unsaponified part of the PVA, and the 2-substitutional benzene was considered as a structure having an amino group at the 0-position on the basis of the chemical shift value. Uncertain peaks are formed at 73.5 ppm, 68.3 ppm, 48.3 ppm and 38.3 ppm, and these peaks are considered as being provided by the structure combining the 2-substitutional benzene and a main chain, though it was substantially impossible to analyze the structure.

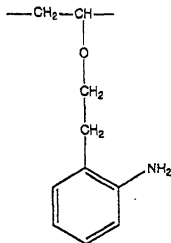
- 2) Chart No. 2 shows results of ^{13}C -NMR (DEPT) measurement on the samples.

The results show that the respective 73.5 ppm and 38.3 ppm in the uncertain peaks of Chart No. 1 denote methylene, 68.3 ppm denotes methine and 48.3 ppm denotes either methine or methyl.

3) Chart No. 3 shows results of ^{13}C -NMR measurement on the methanol-containing samples.

In the measurement, the 48.3 ppm peaks recognized in the above 1) and 2) were consistent with the methanol peak. It was determined accordingly that the 48.3 ppm peak denotes methanol that was included in the original samples in an order of several thousands of $\mu\text{g/g}$.

4) The structure of the part that the main chain and 2-substitutional benzene were combined was estimated as shown below on the basis of the above 1)-3). However, it still remains in the realm of speculation as the chemical shift value cannot be determined.



[Charts]

Chart No. 1-1: ^{13}C -NMR (BCM) measurement result for samples

Chart No. 1-2: Enlarged spectrum of Chart No. 1-1

Chart No. 1-3: Enlarged spectrum of Chart No. 1-1

Chart No. 2-1: ^{13}C -NMR (DEPT) measurement result for samples

Chart No. 2-2: Enlarged spectrum of Chart No. 2-1

Chart No. 3-1: ^{13}C -NMR (BCM) measurement result for samples including methanol

Chart No. 3-2: Enlarged spectrum of Chart No. 3-1

1-1
1-2
1-3
2-1
2-2
3-1
3-2
1-1
1-2
1-3
2-1
2-2
3-1
3-2

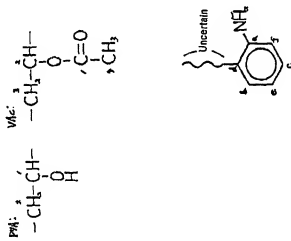
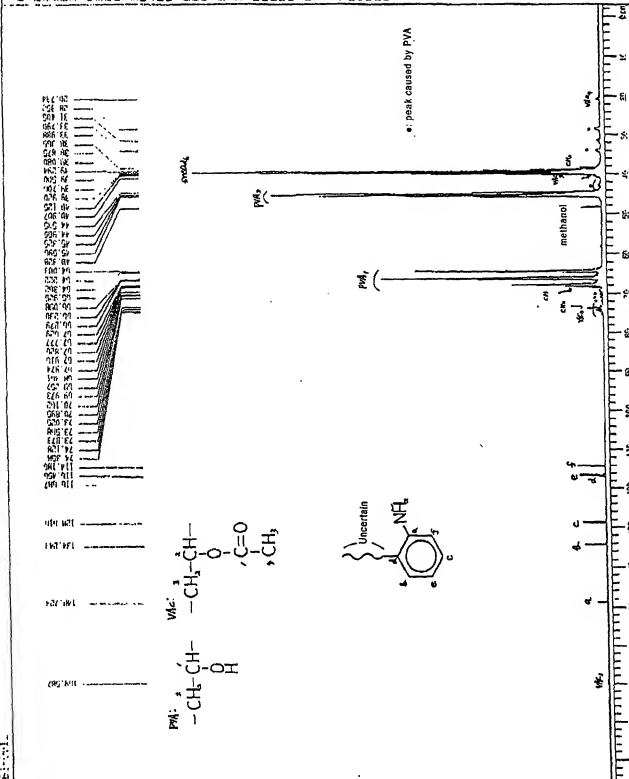


Chart No. 1-1

[illegible]

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ENJOE

• RICHARD ARTHUR ANDERSON
• 61-204
• DEPT.

Year	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099	2100
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$$\begin{array}{cc} \text{CH} & \text{CH}_3 \\ & \uparrow \end{array}$$

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Chart No. 2-1

14 K-
70.0 E

10031-01300 71.000 71.000

Date: Thu Oct 1 10:24:12 1996

File: 10031-01300

Sample: 10031-01300

Method: 10031-01300

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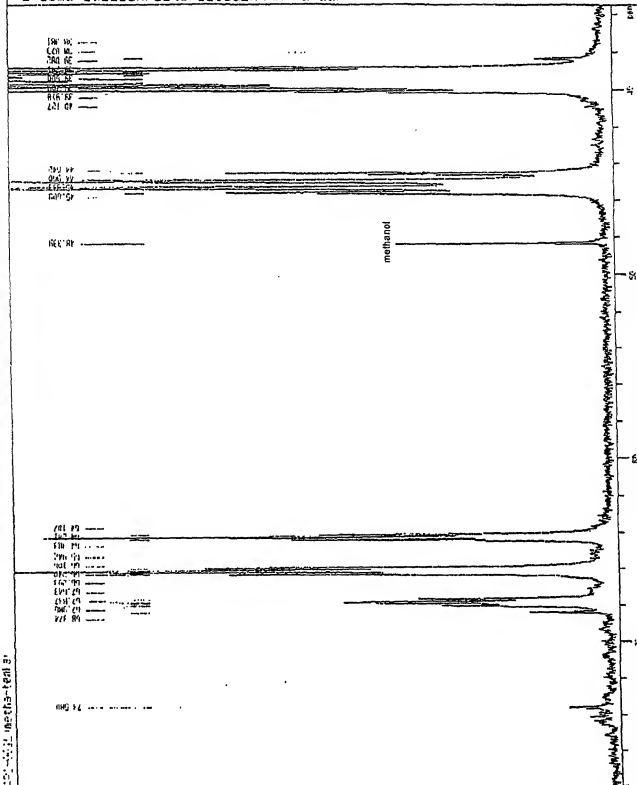


Chart No. 3-2